

LABNOTE

USING THE DECTRIS MYTHEN 1K STRIP DETECTOR – FAST AND HIGH RESOLUTION MEASUREMENTS ON STOE POWDER DIFFRACTOMETERS

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The DECTRIS MYTHEN 1K strip detector has been implemented recently to the STOE powder diffractometer and software. The following examples show to some extent the possibilities of this combination of outstanding equipment for powder diffraction experiments. Extreme fast measurements of typical powder samples are now possible due to the high sensitivity of the MYTHEN 1K detector. A full pattern from 0° to 120° 2θ can be recorded in 60 seconds with a still good signal to noise ratio as shown below.

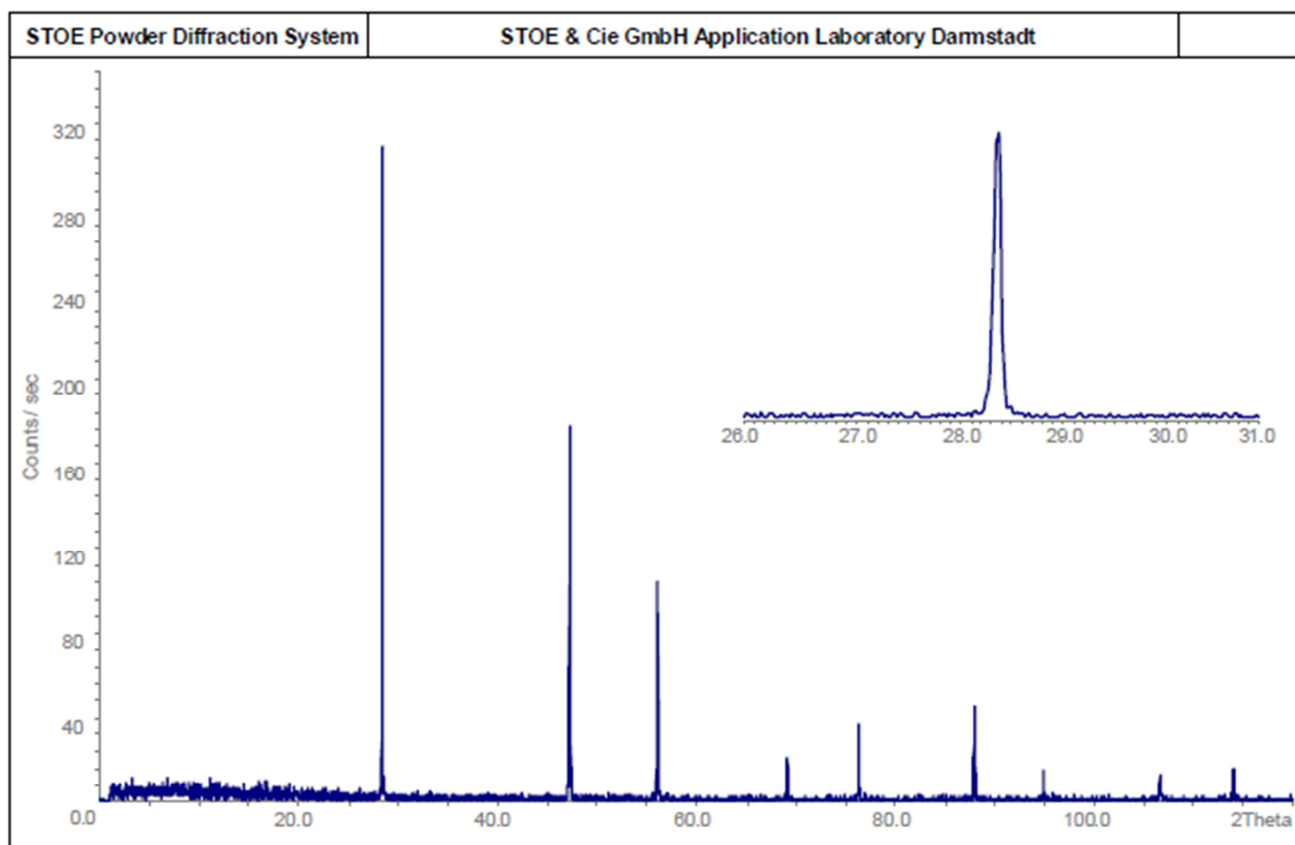


Fig. 1: Measurement of the NIST Silicon standard (NIST SRM 660). STOE StadiP, DECTRIS MYTHEN 1K, Cu- $K_{\alpha 1}$ radiation, transmission sample.

High resolution measurement can be easily performed. A full pattern of a mixture of the three standard materials

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Silicon, Lanthanum Hexaboride and Quartz recorded from 0° to $120^\circ 2\theta$ is shown below.

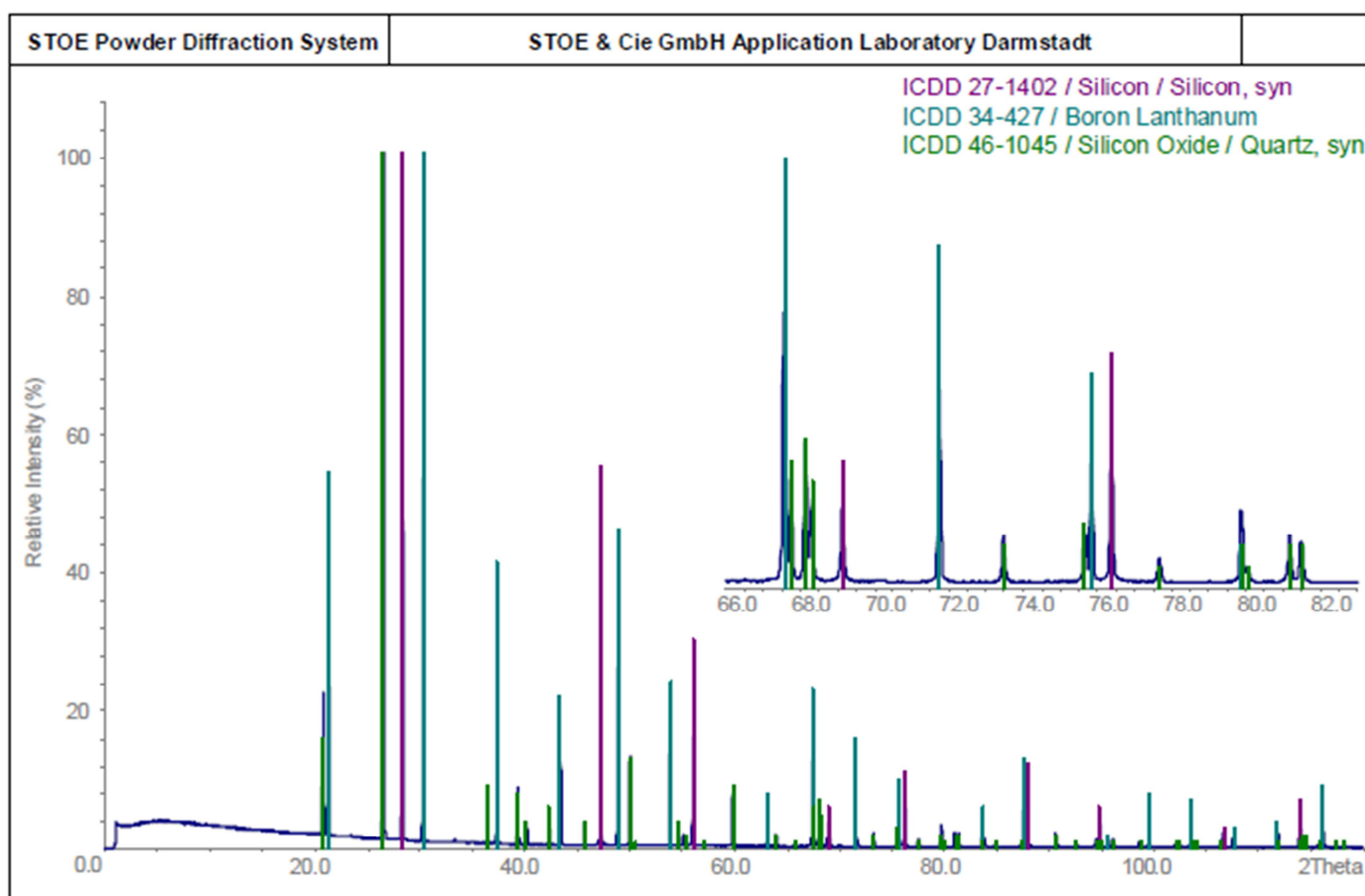


Fig. 2: Measurement of a mixture of Silicon, Lanthanum Hexaboride and Quartz. STOE StadiP, DECTRIS MYTHEN 1K, Cu- $K_{\alpha 1}$ radiation, transmission sample.

The inlay shows a detail of the recorded pattern from 66° to $83^\circ 2\theta$. The (300) reflection of Lanthanum Hexaboride (LaB_6) at 67.56° and the (212) reflection of Quartz (SiO_2) at $67.74^\circ 2\theta$ are as well resolved as the (311) reflection (LaB_6) at 75.85° and the (302) reflection (SiO_2) at $75.76^\circ 2\theta$.

High quality data for Rietveld refinement can also be obtained. A pattern recorded from a sample of Potassium

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Tartrate ($C_4H_5O_6K$, powder ground from single crystals) recorded from 10° to 80° 2θ is shown below. The overlay represents the simulated pattern gained from single crystal structure analysis of the same material.

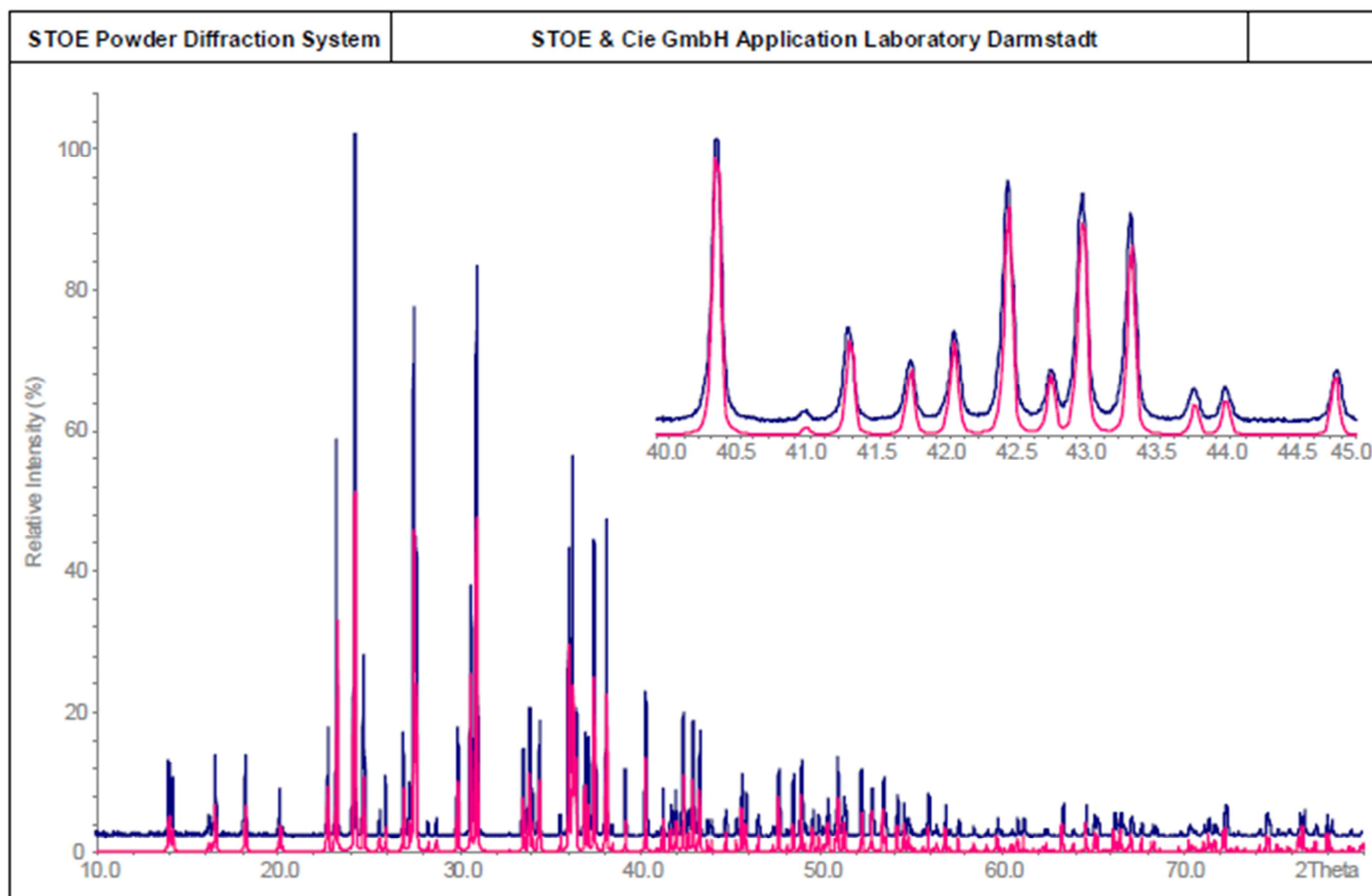


Fig. 3: Measurement of Potassium Tartrate; blue: powder diffraction data; magenta: calculated reflection pattern from single crystal structure analysis (scaled to 50 % intensity for full pattern).
STOE StadiP, DECTRIS MYTHEN 1K, Cu- $K_{\alpha 1}$ radiation, capillary sample.

The inlay shows a detail of the recorded pattern from 40° to 45° 2θ .

The mode of operation of the MYTHEN 1K detector allows the setting of an energy threshold. This enables the suppression of radiation of lower energy. The powder pattern of a 20/80 mixture of Quartz and Iron Oxide with and without the setting of this threshold is shown as an example below.

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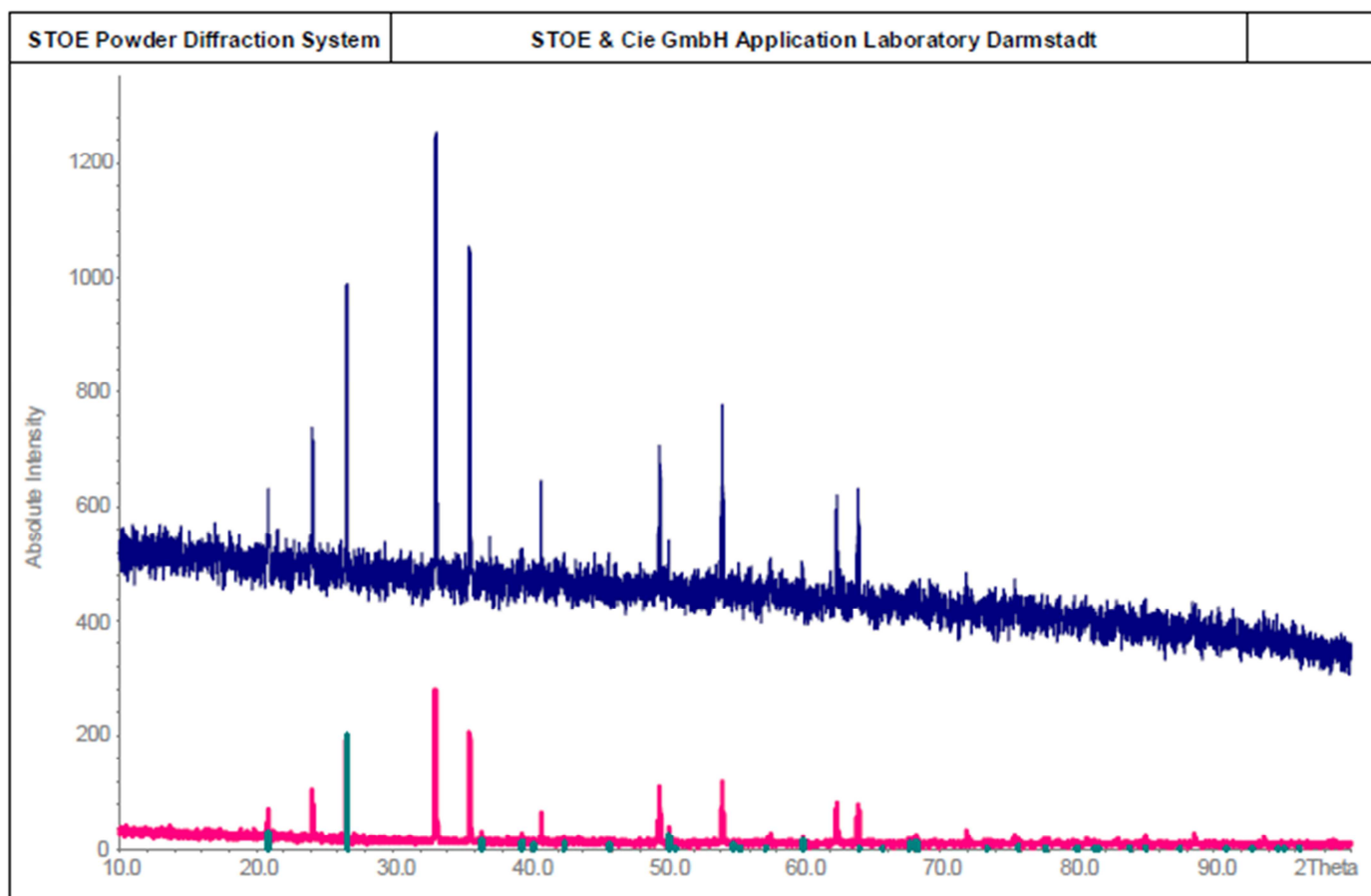


Fig. 4: Measurement of a mixture of Quartz and Iron Oxide; blue: data without the setting of an energy threshold; magenta: data with the setting of an energy threshold; turquoise: ICDD 46-1045 / Silicon Oxide / Quartz, syn; unindexed reflections: Iron Oxide. STOE StadiP, DECTRIS MYTHEN 1K, Cu- $K_{\alpha 1}$ radiation, transmission sample.

Both measurements have been performed with same sampling parameters and can be compared directly. The fluorescence of the Iron Oxide with the used copper $K_{\alpha 1}$ radiation can be suppressed nearly completely while the intensity of the reflection patterns of the Quartz and Iron oxide only decreases about 60 %. Both components can now be indexed.